

EDITORS

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A CITRINE with Eye-Visible Brazil-Law Twinning

The New York laboratory recently received a dark brownish orange pear shape for identification. The stone, which measured approximately $18.70 \times 10.20 \times 7.30$ mm, was set in a yellow metal brooch with numerous variously colored and shaped transparent stones (figure 1). Standard gemological examination and the presence of mineral inclusions established that the stone was natural citrine. Of particular note was the striking eye-visible Brazil-law twinning decorated by cloud-like inclusions (figure 2, left).

Brazil-law twinning is common in amethyst and citrine, but typically it can only be seen by viewing the stone parallel to the optic axis between crossed polarizers. Finding the optic-axis direction in a faceted stone can be challenging because colored gemstones are usually oriented for maximum weight retention and best color, not crystallographically. Thus, Brazil-law twinning is usually seen only after careful observation using a polariscope and an immersion cell (see, e.g., R. Crowningshield et al., "A simple pro-



Figure 1. The dark pear-shaped citrine at the far left in this brooch shows eye-visible Brazil-law twinning with fiber-optic light.

cedure to separate natural from synthetic amethyst on the basis of twinning," Fall 1986 *Gems & Gemology*, pp. 130–139).

However, the twinning in this stone was visible to the unaided eye using only fiber-optic light. Even more unusual, the appearance was stronger with fiber-optic light (again, see figure 2, left)

than between crossed polarizers (figure 2, right). In this case, according to GIA chief gemologist John Koivula, the mounting and the faceted shape of the stone both limit the transmission of polarized light through the citrine, although immersion of the piece (even in water) should result in a clearer polarized light pattern. The fact that the

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Figure 2. The Brazil-law twinning in this citrine is visible to the unaided eye using only fiber-optic light (left); it is also more easily seen with fiber-optic light than between crossed polarizers (right). Field of view ~7.9 mm.

stone was cut with the table perpendicular to the optic axis was also a factor in the ease of observation, as was the presence of a cloud of minute particles trapped in the Brazil twin lamellae.

Wai L. Win

Figure 3. Sponge coral, because of its porous nature, is often impregnated with plastic to strengthen it. Note the continuous pattern of plastic-filled polyp cavities in this 54.88 ct piece.



COMPOSITE OF Coral and Plastic

The Carlsbad laboratory recently began receiving multiple orange beads of various sizes and shapes for identification. These initially appeared to be a plastic-impregnated variety of coral that is often referred to as “sponge coral” because of the numerous natural voids

Figure 4. Unlike the bead in figure 3, this sample (8.65 ct) proved to consist of pieces of coral and other natural materials held together with a plastic binder.



typical of this material. It is a common practice to fill these voids with plastic to strengthen the coral and make it more suitable for jewelry use (figure 3).

Close inspection of these beads revealed, however, that although they contained many of the round- to oval-shaped cavities that are characteristic of sponge coral, there was a lack of continuity in the pattern. There were large areas that had no structure at all and instead were filled with numerous tiny irregular pieces of some other material (figure 4) as well as gas bubbles. It became clear that these samples were in fact a composite of fragments of sponge coral held together with an orange plastic that had been mixed with a ground-up material, probably coral or shell.

To confirm this identification, we soaked a single bead in a bath of methylene chloride for approximately 48 hours. This caused the plastic component to dissolve and the bead to disintegrate into its composite pieces (figure 5).

This is the first time we have encountered such an assemblage with coral, although a similar intent was evident in recent samples of ruby fragments held together with a high-lead-content glass. Therefore, we described the new material on the laboratory report in the same manner, calling it a “coral/plastic composite.”

Kimberly M. Rockwell

Figure 5. After it was soaked in methylene chloride to remove the plastic, the bead in figure 4 disintegrated into fragments.



DIAMOND

Black Diamonds

Colored by Hydrogen Clouds

The vast majority of black diamonds submitted to the laboratory have been treated by high-temperature heating in a vacuum. These stones are usually opaque and heavily fractured. Black graphite inclusions, induced by heating, block light transmission and cause the host diamond to appear black.

Recently at the New York laboratory, we examined two cut-cornered rectangular brilliants, weighing 2.69 and 2.72 ct, that were submitted at the same time. Both were color graded Fancy black (figure 6), but unlike treated black diamonds, these two stones were transparent to fiber-optic illumination and contained numerous dense hydrogen clouds but no evidence of graphite inclusions. A strong 3107 cm^{-1} peak, attributed to hydrogen, was visible in their mid-infrared absorption spectra (figure 7). After nitrogen, hydrogen was the most abundant impurity in these diamonds. The dense hydrogen



Figure 6. The black color of these diamonds (2.69 and 2.72 ct) was caused by dense inclusions of hydrogen clouds.

clouds are responsible for the natural black color.

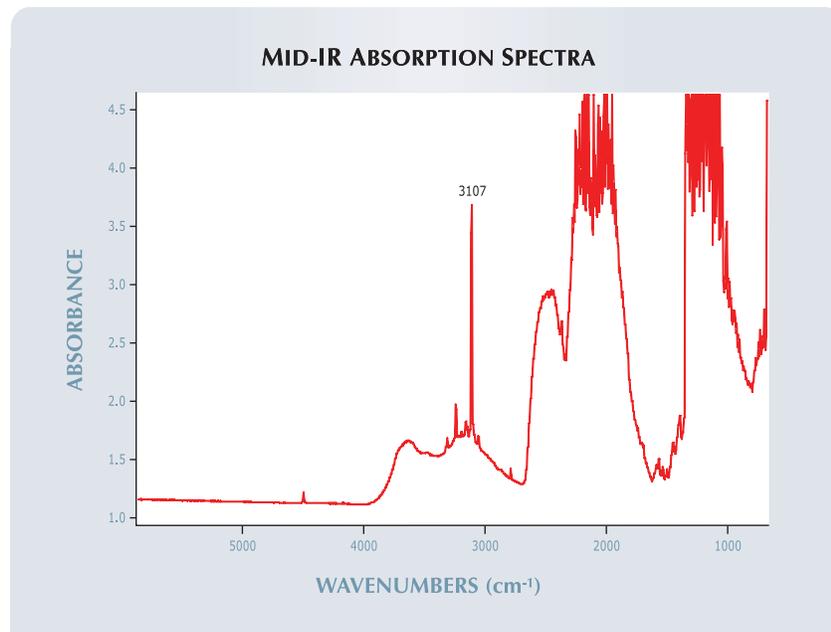
The two diamonds were almost identical, and we suspected that they had originated from the same crystal. This was supported by DiamondView images of the two stones, which showed striking almost-mirror-image butterfly structures in the center of their table facets (figure 8).

The distribution of these symmetrical hydrogen clouds throughout the crystal was dictated by the {100} growth direction of the diamond lattice (W. Wang and W. Mayerson, "Symmetrical clouds in diamond—the hydrogen connection," *Journal of Gemmology*, Vol. 28, No. 3, 2002, pp. 143–152). The tables of both diamonds were cut and oriented in the {100} crystal face direction, resulting in the butterfly images that indicated their origin from the same piece of rough.

The grading reports we issued for these two stones stated that they had a natural origin of color.

Paul Johnson

Figure 7. The mid-IR absorption spectra of the two Fancy black diamonds show a strong 3107 cm^{-1} peak, attributed to hydrogen.



Irradiated Diamond: An Easy Call

Given the complexity and variety of diamond treatments these days, it is not often that the nature of a stone can be decided with a relatively quick look through the microscope. Heat, radiation, and pressure are applied and reapplied to both natural and synthetic diamonds; surface-reaching cracks are filled with glass; surfaces are coated; and the geologic origin indicated by mineral inclusions is negated through the application of focused laser energy and acid boiling. The equipment needed to conclusively identify treatment in diamonds is becoming increasingly



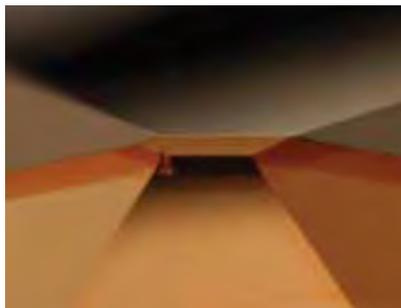
Figure 8. The symmetry of the DiamondView images indicates that the two black diamonds were cut from the same crystal. Butterfly ~3 mm wide.

expensive, highly analytical, and fully computerized.

So, though it does not happen often anymore, it is refreshing to encounter a treated diamond that requires only a microscope and gemological training to determine whether and how it was treated. Recently, a 4.06 ct dark orange-brown, marquise-shaped brilliant provided just such an opportunity.

The stone, which was submitted for origin-of-color determination, measured $17.95 \times 8.09 \times 4.86$ mm. Viewed face-up with the unaided eye, it appeared to have an evenly distributed bodycolor. Viewed through the microscope, however, its story was completely different. The orange-brown color of this type IIa diamond was clearly concentrated along the pavilion facet junctions and around

Figure 9. The obvious orange-brown color zoning along the facet junctions of this cyclotron- and heat-treated diamond makes the treatment determination easy. Field of view ~1.5 mm.



the culet (figure 9), creating a slightly distorted version of the so-called umbrella effect that results from irradiation treatment applied in a cyclotron. Bombardment of a diamond in a cyclotron generally produces an “olive” green color. If such a stone is then heated in air to $590\text{--}620^\circ\text{C}$, the color will change to an orange-brown (J. I. Koivula, *The MicroWorld of Diamonds*, Gemworld International, Northbrook, IL, 2000, pp. 64–65), which presumably is what happened to this diamond. Although decades ago many diamonds were treated in cyclotrons, we rarely encounter them in the laboratory today, probably because detecting the treatment is such an “easy call.”

John I. Koivula and Laura L. Dale

Natural Type Ib Diamond with Unusual Reddish Orange Color

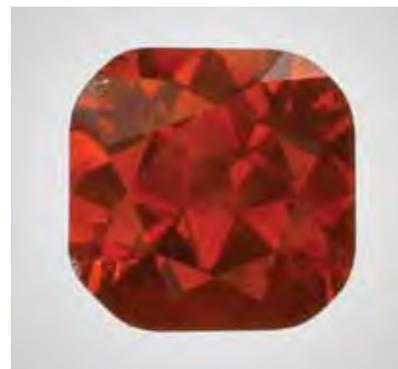
Few natural diamonds contain detectable levels of isolated nitrogen. These stones usually display an orange-yellow color, occasionally with brownish modifiers. The trade refers to some stones in this group as “canary” yellow. In the New York laboratory, however, we recently examined a diamond with isolated nitrogen that was an unusual reddish orange color.

This 1.32 ct round-cornered square brilliant ($6.18 \times 6.14 \times 4.42$ mm) was color graded Fancy reddish orange (figure 10), which is very rare in natural-color diamonds. It was inert to conventional long-wave ultraviolet (UV) radiation, but showed very

weak orange fluorescence to short-wave UV. With magnification, this diamond revealed clouds of tiny particles that spread out across most of the table facet and exhibited both symmetrical and irregular shapes. Clouds are a very common feature in natural type Ib diamonds, and with this exception the color was distributed evenly. The infrared absorption spectrum revealed that this stone contained a substantial amount of isolated nitrogen (34 ppm). In addition, it contained about 11 ppm of the A aggregate of nitrogen. The occurrence of A aggregates contributed to the formation of the H3 defect, which was distributed along several sets of lamellae and exhibited clear green fluorescence in the strong UV radiation of the DiamondView.

The UV-visible–near infrared (UV-Vis-NIR) absorption spectrum (figure 11), collected at liquid-nitrogen temperature, displayed a very smooth absorption curve with no sharp peaks. The absorption increased dramatically from ~620 nm to the high-energy side, with total absorption at wavelengths below 480 nm. This type of absorption pattern could be attributed to a specific concentration of isolated nitrogen, and created a transparent window in

Figure 10. The unusual color of this 1.32 ct type Ib Fancy reddish orange diamond could be attributed to a specific concentration of isolated nitrogen. Reddish hues are very rare for natural type Ib diamonds.



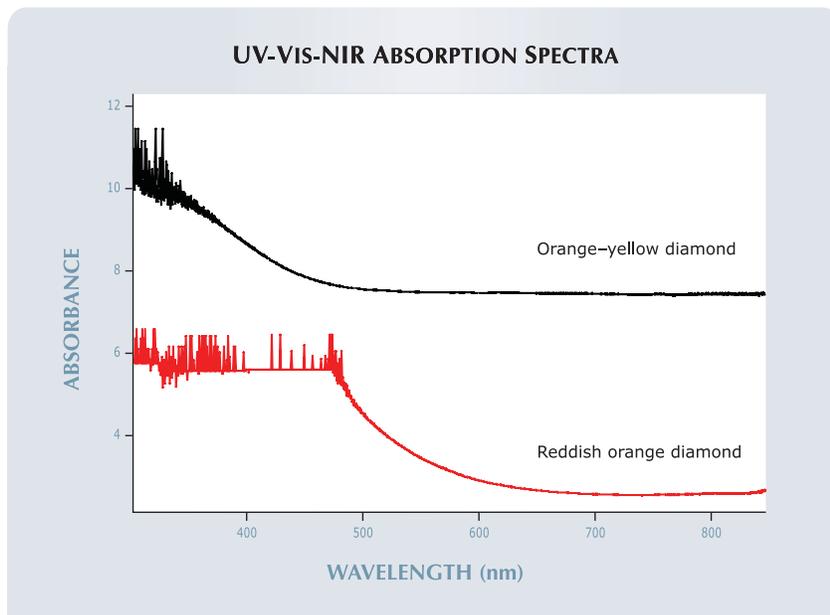


Figure 11. In its UV-Vis-NIR spectrum, the 1.32 ct reddish orange diamond showed much stronger absorption in the yellow-green region than do typical type Ib diamonds colored by isolated nitrogen (e.g., top spectrum). Spectra offset vertically for clarity.

the red-orange region. The color contribution from the H3 defect was trivial, if any. With either higher or lower concentrations of isolated nitrogen, there might not have been a reddish hue.

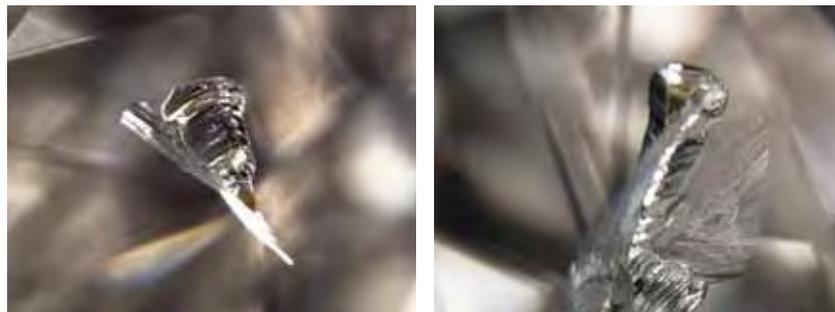
Examination of this special diamond indicated that reddish hues can be present in some rare type Ib diamonds, in addition to the well-known orange-yellow colorations.

Wuyi Wang

Observed Oddities in Diamond

Occasionally, features we observe in diamond are appreciated for their appearance alone rather than any scientific value they might have (e.g., Lab Notes: Winter 2007, pp. 363–364; Summer 2008, pp. 157–158). Olivine is one of the most common inclusions in diamond, and usually no second thought would be given to such an inclusion beyond its effect on the

Figure 12. This olivine inclusion and its associated cleavage fracture (left) resemble a winged insect at high magnification (right). Length of the crystal is ~0.56 mm.



diamond's clarity grade. At the Carlsbad laboratory, however, we recently observed an olivine inclusion in a diamond that was interesting because it had the appearance of a winged insect (figure 12). The anatomy of the crystal was unusual, consisting of a hexagonal cross-section and a bulbous shape that, when combined with the associated cleavage fracture "wings," reinforced the resemblance to a moth or mayfly.

Burned patches on a diamond are likewise nothing out of the ordinary—normally just a surface feature that can affect the polish grade. Recently, though, a stone submitted to the Carlsbad laboratory was observed to have an unusually deep polish feature with a decidedly "tropical" appearance (figure 13). The burn—on an extra facet—resembled flowers or ferns; similar to ferns, it appeared almost fractal in nature. Covering the entire extra facet (approximately 0.55 mm wide), the burn feature had surprising depth of relief, which was especially evident at the facet edges.

These two features show us, once again, that the seemingly mundane task of grading diamonds can prove interesting with the application of a little imagination.

Karen M. Chadwick

Figure 13. The burn on this extra facet resembles ferns or flowers and has surprising depth of relief, readily seen at the facet edges. Field of view ~0.7 mm.





Figure 14. Discovered in a brownish green type Ia diamond, this hydrogen cloud has a unique three-dimensional shape reminiscent of a Venetian party mask. Field of view ~2.9 mm.

“Party” Diamond

In past Lab Notes sections, we have reported on a variety of cloud patterns in diamonds (e.g., Spring 1999, pp. 42–43; Fall 2000, pp. 255–256; Spring 2001, pp. 58–59). Cloud formations in diamonds are actually phantoms mimicking the growth of their hosts. As such, most clouds have stellate or cruciform habits that can be described geometrically by the isometric crystal system that governs the structure of single-crystal diamonds. Of course, there are exceptions to every rule, and the hydrogen cloud pictured in figure 14 definitely fell into that category.

As is typical with hydrogen clouds, this one was hosted by an off-color type Ia diamond, in this case a dull brownish green round brilliant cut that was submitted to the Carlsbad lab for an origin-of-color determination. While this was a routine submission, the pattern shown by the more-or-less centrally located phantom cloud was anything but routine. When examined with fiber-optic illumination from the side, the cloud

had the appearance of a Venetian mask, similar to those worn by partygoers at a masquerade ball. Given the cloud’s unique and complex appearance, it was impossible to clearly describe its geometry in terms of the known crystal habits of diamond (see, e.g., V. Goldschmidt, *Atlas der Kristallformen*, Vol. 3, C. Winters, Heidelberg, Germany, 1916).

While we thought we recognized both octahedral and dodecahedral structural components, the “eye” holes in the mask-shaped cloud—which were devoid of light-scattering, cloud-forming particles—did not seem to fit either morphology. This cloud’s appearance was so unusual in our experience that we felt it worthy of documentation, even though a geometric description was not possible.

John I. Koivula and Laura L. Dale

Pinkish Brown Diamond with Mantle-Mineral Inclusions

Among natural purple-to-pink diamonds, some are distinguished by color

color concentrations along octahedral {111} glide planes. When the colored glide planes are positioned properly, the otherwise subtle pink hue is intensified. Our observations have revealed that almost all purple-to-pink diamonds with glide planes are type IaA with moderate-to-high concentrations of nitrogen, which indicates a correlation between the formation of glide planes and the nitrogen aggregation state. In the New York laboratory, we recently examined a large diamond of this type. Gemological and spectroscopic analysis revealed some interesting features, including unique mantle-mineral inclusions.

The 4.29 ct diamond was color graded Fancy pinkish brown (figure 15). It had an unusual cutting style: a modified heart brilliant with a wavy top edge in place of the usual lobes. It fluoresced moderate blue to long-wave UV radiation and very weak yellow to short-wave UV. With magnification, sharp parallel glide planes with concentrated pink color were easily observed throughout most of the stone. The strong UV radiation of the DiamondView revealed linear green fluorescence perfectly aligned along the glide planes due to localized

Figure 15. The color of this 4.29 ct Fancy pinkish brown diamond is caused by a broad absorption band at ~550 nm related to internal glide planes. It is also notable for its unusual cutting style and mineral inclusions.

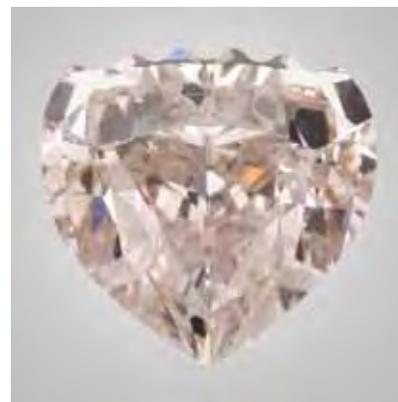




Figure 16. This large octahedron (~1.0 mm long) seen in the pinkish brown diamond is believed to be chromite. The parallel lines on its surface very likely formed simultaneously with the glide planes in the host.

formation of H3 defects. As expected, the infrared and UV-Vis absorption spectra showed that the diamond was type IaA with a high concentration of nitrogen, and that it was colored by absorption at ~550 nm.

An outstanding feature of this diamond was its inclusion of minerals from the earth's mantle. Colorless euhedral inclusions of varying sizes were confirmed to be olivine by Raman spectroscopy. In addition, a large black octahedron was present at the top of the wavy region (figure 16). Based on its color, luster, and morphology, it was probably chromite, though we could not confirm this with Raman analysis. An interesting feature of this inclusion was the presence of parallel lines on its surface (again, see figure 16), which very likely formed simultaneously with the glide planes in the host diamond. This is the first time we have observed glide planes in both the host diamond and its chromite inclusion.

Wuyi Wang

Large EMERALD-in-Quartz Specimen

Laboratory gemologists deal with the identification of treatments, synthetics, and imitations on an almost rou-

tine basis. So when the opportunity to examine a truly rare natural gem material arises, it is greeted with a good deal of enthusiasm. Such was the case recently, when we examined a large transparent quartz crystal that contained eye-visible, gem-quality inclusions of emerald (figure 17), a first in our experience. An intergrowth of emerald and quartz was reported in the Summer 2000 Lab Notes section (pp. 164–165), but that faceted stone was essentially half semitransparent white quartz and half low-quality emerald—quite different from the specimen described here.

The 69 g quartz crystal (74.05 × 31.91 × 20.41 mm) was reported by emerald dealer Ron Ringsrud (Ronald Ringsrud Co., Saratoga, California) to have been recovered from the La Pita mine in Boyacá, Colombia. He submitted the specimen for gemological examination with the permission of

Figure 17. This 74.05-mm-long specimen of emerald in quartz was recovered from the La Pita mine in Boyacá, Colombia.



the owner, German Sanchez (Universal Emerald CI Ltda., Bogotá).

As seen in figure 17, the quartz crystal was well formed, singly terminated, and highly transparent, making the emerald inclusions clearly visible without magnification. A large cluster of emerald crystals reached the surface of the host quartz, which indicates they formed first and then the quartz grew over them. The largest of the inclusions was 23.50 mm long (figure 18), so an emerald spectrum was easily obtained when it was examined in transmitted white light with a handheld prism spectroscope. This is one of the most unusual “inclusion” specimens we have ever seen.

*John I. Koivula
and Shane F. McClure*

Figure 18. The largest of the inclusions (23.50 mm long) in the quartz crystal revealed a characteristic emerald spectrum when examined with a handheld spectroscope.



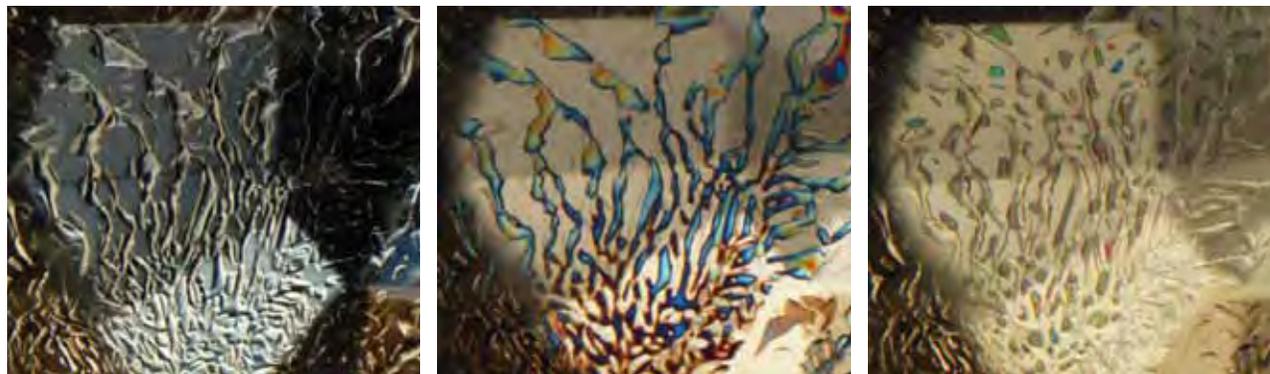


Figure 19. Raman analysis proved that this fluid-appearing layer of transparent inclusions in a grossular reportedly from Tanzania (left) was calcite. In one orientation in polarized light (center), some of the calcite inclusions display interference colors, while others do not. Upon 90° rotation of both polarizer and analyzer (right), the previously extinct portions now show interference colors, while the previously bright areas do not. Field of view 1.8 mm.

Calcite “Melt” in GROSSULAR

In the past, we have seen unusual-looking transparent features in pale-colored grossular from Lelatema, Tanzania, that appeared as if they might have been molten at the time they were included in their garnet hosts. Although those inclusions were examined microscopically, and under polarized light proved to be solid and birefringent, on these previous occasions we did not have the opportunity or capability to identify them.

Recently, we encountered a 0.39 ct transparent light green, round mixed-cut grossular—reportedly from Tanzania—with inclusions that were very similar to those we had previously observed. As shown in figure 19 (left), this feature was a transparent dendritic or fan-shaped layer of fluid-like inclusions that gave the strong appearance of once having been molten.

The pale green garnet host had the expected RI of 1.731. It exhibited a weak-to-moderate orange reaction to long-wave UV radiation, while the inclusion layer was inert. In polarized light, the transparent areas of the inclusion system behaved curiously. In one orientation of the polarizer and analyzer, a portion of the system showed bright interference colors while the remainder was extinct (fig-

ure 19, center). When the polarizer and analyzer were rotated in unison by 90°, the previously extinct area showed bright interference colors, while the previously bright portion went extinct (figure 19, right). This clearly revealed that *two* crystallographic orientations were present within this system. Examining all three photomicrographs side by side as a triptych made this curious relationship quite obvious.

We then employed laser Raman microspectroscopy to determine the nature of this feature. The results clearly showed that the melted-looking inclusions were calcite. Calcite is known to occur in garnets from this and other localities (such as hessonite from Sri Lanka), but it has not previously been reported with such a “liquid” form. An interesting bonus in this investigation was that a tiny, nondescript, rounded inclusion near the girdle and the pavilion surface, which was also analyzed, proved to be elemental sulfur, though it was too small to show its characteristic yellow color. Sulfur has not been encountered before as an inclusion in garnet. The investigation of this grossular showcases the power and increasing usefulness of Raman analysis in inclusion studies.

John I. Koivula and Alethea Inns

A Rare Yellow Trapiche SAPPHIRE

The 40.57 ct transparent-to-semi-transparent yellow oval tablet in figure 20 was submitted to the New York laboratory as a “star sapphire.” Standard gemological testing identified it as a natural sapphire. What caught our eye immediately, though, was that the star-like appearance was not asterism, but rather a “trapiche”

Figure 20. This 40.57 ct yellow sapphire (21.60 × 16.30 × 8.60 mm) exhibits the trapiche growth phenomenon.





Figure 21. The core of the trapiche crystal is visible at upper left, with the arms formed by radiating spokes containing ribbons of thin films. Field of view ~3 mm.

pattern: a fixed reflective six-armed star with associated color zoning.

While trapiche patterns are common in emerald, they are less common in corundum, in which they are typically restricted to ruby (see, e.g., K. Schmetzer et al., "Trapiche rubies," Winter 1996 *Gems & Gemology*, pp. 242–250; I. Sunagawa et al., "Texture formation and element partitioning in trapiche ruby," *Journal of Crystal Growth*, Vol. 206, No. 4, 1999, pp. 322–330). Trapiche sapphire is considerably rarer (see, e.g., box A of T. Hainschwang et al., "Trapiche tourmaline from Zambia," Spring 2007 *Gems & Gemology*, pp. 36–46), and

most is blue to gray: An extensive literature search with the help of staff members at GIA's Liddicoat gemological library revealed no published accounts of yellow trapiche sapphire.

Exposure to long-wave UV radiation caused this stone to fluoresce weak-to-moderate orange in the triangular growth sectors, while the "arms" fluoresced weak orange or were inert; the short-wave UV reaction was weaker. Fluorescent Cr lines at ~680 nm and an absorption line at 450 nm in the desk-model spectroscope indicated the presence of trace amounts of Cr and Fe, respectively.

A small core area parallel to the c-axis formed the center of the star formation. Reflected light best revealed the inclusions. The triangular growth sectors had a yellow bodycolor and were less included—with mostly fluid remnant "fingerprints," two-phase inclusions, and reflective films—than the radiating spokes. The arms were nearly colorless and to the unaided eye appeared to contain abundant needles. With magnification, these "needles" could be resolved into narrow ribbons of reflective thin-film inclusions in planes that were approximately perpendicular to the core or c-axis (figure 21). Metallic, small dark, or irregular transparent crystals were present in both the arms and triangular growth sectors.

These differences in inclusions, color, and growth structure suggest that the sapphire crystallized from its core outward, with the sectors and arms

probably growing concurrently but at different rates. Note that the definition of *trapiche* varies, with some referring to it as skeletal growth resulting in inclusions trapped in the arms of the star, or being composed of a different material than the host (Hainschwang et al., 2007). Other definitions include preferential exsolution of inclusions in the dendritic and growth sectors, or structure- and growth-related chemical variations that result in color-causing mechanisms differentiating the arms (K. K. Win, "Trapiche of Myanmar," *Australian Gemmologist*, Vol. 22, No. 6, 2005, pp. 269–270). Although many of the reported trapiche corundums—and rubies in particular—have arms composed of distinct mineral phases, this stone was primarily composed of corundum throughout. Even so, the GIA Laboratory categorized this sapphire as "trapiche" due to its distinct core, inclusion pattern, and growth structure. It is the first yellow trapiche sapphire we have seen.

Donna Beaton

PHOTO CREDITS

Jian Xin (Jae) Liao—Figures 1, 6, 15, and 20; Wai L. Win—Figure 2; C. D. Mengason—Figures 3, 4, and 5; Paul Johnson—Figure 8; John I. Koivula—Figures 9, 12 (left), 14, and 19; Elizabeth Schrader—Figure 10; Karen M. Chadwick—Figures 12 (right) and 13; Wuyi Wang—Figure 16; Robert Weldon—Figures 17 and 18; Donna Beaton—Figure 21.

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